

## North Dakota Renewable Energy Program Status Report

Recipient: Xiaodong Hou University of North Dakota/Institute for Energy Studies  
Contract Number: R-035-044  
Report for time period of: 09/01/2019-3/31/2020

### No-cost Extension Request

We requested a three-month no-cost extension because of the delayed equipment purchase process. The request was approved by NDIC. The new deadline of the project is May 31, 2020 with a final report due on June 30, 2020. Therefore, this is the 4<sup>th</sup> and an additional semi-annual report stated on the updated agreement.

### Description of Project

There is no change on the project goals and objectives because of no-cost extension. The overall goal of this project is to develop a low-cost synthetic procedure to prepare graphene-modified LiFePO<sub>4</sub> (LFP/G) as a high-performance cathode material for LIBs at pilot scale (10 tons/year). To fulfill this goal, a two-step procedure is proposed: 1) humic acid is extracted and purified from low-rank ND coal or leonardite and 2) using the extracted humic acid as a key feedstock to in situ prepare LFP/G via a novel modified carbothermal reduction reaction.

### Project Tasks

Please describe the progress on all project tasks achieved during the reporting period:

Five tasks have been defined to fulfill the above goals. In the past six months, the project is focused on the task 4 and 5 as planned in the timelines along with the few unfinished items on Task 2 and 3 from the previous report period. The progress is outlined as follows:

1. Task 2, optimization of LFP/G synthesis procedure is completed.
  - The only unfinished parameter, the feeding ratio of humic acid is optimized.
  - Coin cells prepared with LFP/G DOE have exhibited high performance.
2. Task 3, characterization of LFP/G cathode results:
  - Crystalline purity by XRD, carbon content by TOC analyzer, tap density and Morphology analysis by SEM were completed for majority of samples. All the above tests will be performed on the optimal sample. Raman spectroscopy to be run on LFP/G samples to verify graphene formation.
  - An optimal carbon content is defined by adjusting the humic acid feeding ratio.
3. Task 4, Pilot scale production has proven to be scalable based on optimized extraction procedures at lab scale.
  - A total of 8 kg humic acid (dry basis) was produced at one batch by the pilot scale facility.
  - The preparation of LFP/G at scale of Kilogram-level is on-going.
  - Purity – Ion Exchange – Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> Sodium Paraphosphate
4. Task 5, electrochemical performance testing is currently being conducted on cell prepared with our LFP/G cathode material.

- The focus is on the cells prepared by the optimal procedure. We expected they will exhibit even better performance than our preliminary results and also much better than that of commercial LFP/C.
- The specific capacity of the optimal cathode material is close to the target value 150 mAh/g. Further refinement is in process.

**Deliverables**

Please describe the progress on project deliverables, as stated in your contract. Achieved during the reporting period:

- Completion the optimization of all the parameters for LFP/G synthesis.
- Pilot scale humic acid (8 kg), after purification steps, shown to be viable for high-performance batteries.
- 15% specific capacity improvement over commercial products (120-125 mAh/g) and better cycle life.

To be achieved soon:

- 1 Kg pilot scale production of LFP/G.
- Battery performance test on larger cells.
- Final report.

Deliverables achieved during the previous period:

- Development of the procedure that can produce high-purity (>99%) and metal-free humic acid from leonardite. We have achieved the high purity (ash <1%) and low iron content of (0.2%) humic acid derived from leonardite.

**Expenditures**

Please provide a breakdown of expenditures. Include all sources of match. Provide supporting documentation as a separate attachment.

| EXPENDITURES FOR THIS REPORTING PERIOD ONLY |                  |                  |                  |                   |
|---|------------------|------------------|------------------|-------------------|
| Project Expense                             | NDIC             | REP Recipient    | Other Sponsor    | Total             |
| Salary & Fringes                            | 30,113.22        | 29,985.07        | 24,064.00        | 84,162.29         |
| Supplies                                    | 11,415.55        |                  | 237.00           | 11,652.55         |
| Fees  | 5,500.46         |                  | 30,666.00        | 36,166.46         |
| Indirects                                   | 16,133.20        | 11,693.73        | 13,741.00        | 41,567.93         |
| <b>Total</b>                                | <b>63,162.43</b> | <b>41,678.80</b> | <b>68,708.00</b> | <b>173,549.23</b> |

| CUMULATIVE EXPENDITURES |                   |                  |                   |                   |
|-------------------------|-------------------|------------------|-------------------|-------------------|
| Project Expense         | NDIC              | REP Recipient    | Other Sponsor     | Total             |
| Salary & Fringes        | 115,038.76        | 61,250.00        | 142,408.00        | 318,696.76        |
| Supplies                | 25,655.61         |                  | 19,237.00         | 44,892.61         |
| Fees                    | 23,286.56         |                  | 66,803.00         | 90,089.56         |
| Indirects               | 61,744.79         | 23,887.00        | 46,833.00         | 132,464.79        |
| <b>Total</b>            | <b>225,725.72</b> | <b>85,137.00</b> | <b>275,281.00</b> | <b>586,143.72</b> |